

Manure Phosphorus Extractability as Affected by Aluminum- and Iron By-Products and Aerobic Composting

Thanh H. Dao,* L. J. Sikora, A. Hamasaki, and R. L. Chaney

ABSTRACT

Shifts in manure phosphorus (P) chemical forms and pool sizes induced by water treatment residuals and industrial mineral by-products are largely undefined. We conducted a manure P fractionation study to determine mechanisms of reduction of dissolved reactive phosphorus (DRP) in poultry manure upon mineral by-product additions. The effects of composting on the P immobilization efficacy of the by-products were determined using laboratory self-heating composting simulators. The mineral by-products included an aluminum-water treatment residual (Al-WTR) and an iron-rich titanium-processing by-product. The noncomposted manure averaged 0.11 g g^{-1} of total P as DRP forms. The by-products significantly reduced manure DRP, by an average of 39 and 48% in the Al- and the Fe-treated manure, respectively. The by-products also reduced the $0.5 \text{ M NH}_4\text{F}$ -extractable phosphorus (FEP) fraction. Shifts in P forms between FEP and 0.1 M NaOH -extractable phosphorus (SHEP) depended upon the Al and Fe contents of the by-products while the combined FEP + SHEP pool remained constant. Phosphate sorption measurements supported the observations that the Fe-rich by-product was more effective at reducing manure DRP and enhancing the formation of SHEP forms at the expense of FEP than the Al-WTR. Composting had no effect on the efficacy of either by-product to reduce DRP. Potential mechanisms of enhanced P stabilization in treated manure upon composting included chemical shifts from the DRP and FEP fractions to the citrate-bicarbonate-dithionite extractable P fraction. Thus, the choice of P immobilization agents affected the stability of immobilized P forms and should be taken into consideration in developing manure processing and nutrient stabilization methods.

THE greatest potential for eutrophication of surface waters usually occurs in watersheds with intensive animal production (Council for Agricultural Science and Technology, 1996). Outbreaks of *Escherichia coli* and *Pfiesteria* that have killed fish and caused human illness are thought to result from the contamination of streams, reservoirs, and estuaries by animal manure organisms and nutrients, mainly nitrogen (N) and P (Burkholder et al., 1992). Elevated P and N levels often are linked to algal blooms and accelerated eutrophication of lakes and streams (Levine and Schindler, 1989; Wood and Hattey, 1995; Correll, 1997). Therefore, the beneficial effects of nutrient recycling and long-term manure applications on crop production fields have to be weighed against the detrimental effects of potential offsite discharges on the environment. Although highly variable, the fertilizer value of manure depends upon the frequency of cleaning of the poultry house that is linked to the number of batches of birds grown on the same litter (Xu and Prato,

1995). Land application of such materials can release high loads of soluble and organic P, which may result in potential water quality impairment. Runoff DRP concentrations have been related to surface soil extractable P concentrations (Sharpley et al., 1993; Schreve et al., 1995; Sharpley, 1995).

Nonhazardous municipal and industrial by-products potentially reduce excessive amounts of DRP in animal manure and from manure-amended soils (Schreve et al., 1995; Peters and Basta, 1996; Dao, 1999). Addition of reagent-grade FeCl_3 , FeSO_4 , and alum ($\text{Al}_2[\text{SO}_4]_3 \cdot 14\text{H}_2\text{O}$), a water-soluble salt that released Al^{3+} , has been shown to reduce P solubility in poultry manure (Moore and Miller, 1994). The Al^{3+} reacted with ammonia released during the decomposition of uric acid and organic N in poultry manure. Amorphous aluminum hydroxides formed in alum-treated manure bind and immobilize soluble manure P. Spent residuals from water treatment plants also sorb and precipitate soluble manure P (Peters and Basta, 1996). Unlike alum or aluminum chloride used in the treatment of wastewater, the Al in Al-WTR occurs as amorphous insoluble aluminum oxides and is less likely to form acidity when amended to manure or manure-amended soils. An iron-rich by-product of the titanium oxide extraction process from Dupont Chemicals (Wilmington, DE) has been evaluated as a component of a synthetic soil for the nursery industry (Goyette, 1992). The by-product contained significant levels of iron oxides, other metals such as chromium, zinc, nickel, lead, and chloride, and has been used to reduce dissolved P in poultry manure and manure-amended soils (Salingar et al., 1994; Codling et al., 2000). At application rates between 25 and 50 g kg^{-1} , the iron-rich by-product reduced dissolved manure P by about 40%. Calcium-, Al-, and Fe-rich by-products such as caliche, alum, and Class C coal combustion ash used as a manure amendment reduced water and plant-extractable P in cattle manure at rates of 0.1 g g^{-1} and higher and widened the effective N to P ratio of treated manure by factors ranging from 1.5 to 18 (Dao, 1999). Coal combustion by-products have been applied as a liming material and suggested as a soil amendment to reduce desorbable P from high-P soils (Stout et al., 1998). Fluidized bed combustion fly ash and flue gas desulfurization by-products significantly reduced water-extractable P and Mehlich III-extractable P in the 10 to 80 g kg^{-1} range, immobilizing P in more insoluble Ca-, Al-, or Fe-bound compounds. The Ca^{2+} in these by-products was

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Published in J. Environ. Qual. 30:1693–1698 (2001).

Abbreviations: Al-WTR, aluminum-water treatment residual; CBDP, citrate-bicarbonate-dithionite extractable phosphorus; DRP, dissolved reactive phosphorus; FEP, $0.5 \text{ M NH}_4\text{F}$ -extractable phosphorus; $\text{H}_2\text{SO}_4\text{-P}$, $0.25 \text{ M H}_2\text{SO}_4$ -extractable phosphorus; SHEP, 0.1 M NaOH -extractable phosphorus.

shown to be an economical and safe amendment to reduce the solubility of soil P (Adriano et al., 1978).

Methods for fractioning soil P have been developed to study the transformation of applied P fertilizer and develop a basic understanding of P status and availability in soils. The procedure developed by Chang and Jackson (1957) was proposed to distinguish the Ca-, Al-, and Fe-phosphate fractions and has been modified for different soil types. Hedley and co-workers (1982) proposed acid and base treatments to differentiate soil P pools. Modifications of the Hedley sequential extraction procedure have been used to determine P forms in poultry, swine, and cattle manure (Dou et al., 2000; Sharpley and Moyer, 2000). Manure total P ranged from 2.6 to 40 g kg⁻¹, where 60 to 90% existed as inorganic P and 0.8 to 3.9 g kg⁻¹ as organic P forms. The proportion of inorganic and organic P varied among animal species and manure type. In poultry manure, about 25 to 49% of the inorganic P was water-extractable P, 20 to 35% was bicarbonate-extractable, and 25 to 30% was acid-extractable. The organic P pool averaged 15% and could be as high as 25% of total P in dairy manure.

Composting of animal manure results in 30 to 50% reductions in mass and a material more uniform in nutrient composition. Manure N is stabilized through microbial assimilation and humification. The C to N ratio of the manure may be adjusted to conserve manure N during the composting process (DeLuca and DeLuca, 1997). Many C sources, such as wood chips, shavings, sawdust, and crop residues have been used to adjust the C to N ratio of the mixture. Much attention has been devoted to measurements of the N fraction and its net mineralization to predict the N-supplying capacity of composted manure to meet crop needs (Hadas and Portnoy, 1994; Sikora and Enkiri, 1999). Mineralization of labile C fractions resulted in increased P concentrations in composted manure because P is conserved, as opposed to N that is lost by volatilization. Information on manure P extractability after composting is more limited, particularly for chemically treated manure (Traore et al., 1999; Dao, 1999). The objective of this study was to determine the potential mechanisms of DRP reduction in manure treated with Al-WTR and Fe-rich mineral by-products and the effect of aerobic composting on the efficacy of P immobilization by the by-products.

MATERIALS AND METHODS

Bulk samples of poultry manure were collected from grow-out houses containing 20- to 26-wk-old turkeys. A bulking

agent, wood chips, was added to the manure at the rate of 1:2 by volume of manure to wood chips to increase the C to N ratio and immobilize and conserve manure N upon composting. Two mineral amendments were added to the manure. An Al-water treatment residual was obtained from the Potomac Drinking Water Treatment Plant in Rockville, MD. A granular iron-rich by-product of the titanium oxide production process was obtained from Dupont Chemicals (Wilmington, DE). The by-product, produced in large quantities (5×10^4 Mg) annually in the mid-Atlantic region of the USA contained high levels of iron and chromium (III) (Salingar et al., 1994; Codling et al., 2000). Selected properties of the by-products are presented in Table 1. Total elemental concentrations for Al, Fe, and selected cations were determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) following HNO₃-HCl digestion (American Public Health Association, 1989). The by-products were added to the manure on a 2 to 1 molar basis of total Al or Fe to manure total P (after addition of bulking agent). We assumed that only about half of the spent Al-WTR total Al and the titanium-processing by-product total Fe present may react with the manure P to form the corresponding metal phosphates. The amendment rate of by-products was 590 g Al-WTR kg⁻¹ manure that also contained 16.3 g Fe, and 200 g Fe-rich by-product kg⁻¹ manure that also contained 2.4 g Al.

The untreated and Al- and Fe-treated manure were subdivided and composted in triplicate laboratory self-heating composting simulators. Details of composting conditions can be found in previous work (Sikora et al., 1983). The simulator chambers were weighed before and after the composting to determine mass losses. At the end of the composting period, as indicated by the drop in composting chamber temperature, triplicate samples of each treatment were taken for moisture determinations. Manure subsamples (0.5 g dry wt basis) were digested with concentrated H₂SO₄ for determination of total P content. Selected mineral P fractions were determined using a sequential extraction procedure used by Chang and Jackson (1957), modified by Williams et al. (1980). In summary, triplicate 3-g samples (dry weight basis) with 60 mL 1 M NH₄Cl were weighed into plastic centrifuge tubes and agitated on a gyratory shaker for 30 min. The samples were centrifuged at $10\,000 \times g$ and the supernatant decanted, passed through a 0.45- μ m pore size filter, acidified with concentrated HCl, made to volume in 100-mL volumetric flasks, and stored at 4°C until analysis for dissolved reactive phosphorus (DRP) that was extracted by the salt solution. Sixty milliliters of 0.5 M NH₄F were added to the residue remaining in centrifuge tubes. The residue was resuspended and shaken for 60 min. The mixture was centrifuged and the supernatant decanted. The residue was resuspended with two 15-mL aliquots of saturated NaCl solution, and the two washings added to the NH₄F extract. After filtration and acidification the extract was diluted to 100 mL volume and stored at 4°C until P analysis. Sixty milliliters of 0.1 M NaOH were added to the residue remaining in centri-

Table 1. Selected characteristics of poultry manure and the mineral by-products.

Material	P	Ca	Mg	Al	Fe	Mn
	g kg ⁻¹					
Manure	18.8 (± 0.96) [†]	34.9 (± 5.5) [‡]	3.9 (± 0.5)	—§	0.9 (± 0.1)	0.3 (± 0.03)
Al-WTR¶	2.1 (± 0.036)	3.8	3.8	55.8	27.9	1.9
Fe-rich by-product	1.9 (± 0.003)	—§	—§	11.9	335.0	12.3

[†] Mean (\pm standard deviation) ($n = 3$).

[‡] Total elemental composition after HNO₃-HCl digestion.

§ No data available.

¶ Aluminum-water treatment residual.

fuge tubes. The residue was resuspended, shaken, centrifuged, washed, and the extract prepared as with the previous NH_4F fraction. The residue in centrifuge tubes was resuspended for a third time in 50 mL of 0.3 M $\text{Na}_2\text{C}_6\text{H}_5\text{O}_7$ and 5 mL of 1 M NaHCO_3 . The mixture was heated at 85°C. One gram of sodium dithionite was added, and the mixture was stirred continually for 15 min. The suspension was centrifuged at $10\,000 \times g$ and the supernatant decanted into 100-mL volumetric flasks. A saturated NaCl solution was used to wash the residues and the washings were combined with the supernatant. The residue was resuspended for a fourth time in 60 mL of 0.25 M H_2SO_4 on a gyratory shaker for 60 min. The mixture was centrifuged, and the supernatant was decanted and combined with the NaCl washing of the residue pellets. The P concentration of each extracted fraction was determined by the Murphy and Riley (1962) method on a Lachat Instruments automated ion analyzer (Model 8000; Zellweger Analytics, Milwaukee, WI).

To estimate the P sorption capacity of the mineral by-products, triplicate 2-g samples were weighed into plastic test tubes containing 20 mL of the following standard solutions of $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$ (0, 0.08, 0.16, 0.32, 0.65, 0.97, 1.61, 3.26, 6.45, and 12.90 mmol P L^{-1}). The mixtures of P solution and by-product and triplicate tubes containing P solutions alone were agitated on an end-over-end shaker for 16 h. After centrifugation at $10\,000 \times g$ for 20 min, the supernatant solutions were filtered and P concentration determined by the Murphy and Riley method as described above. The amounts of P sorbed by the mineral by-product were calculated as the difference between the amounts of P in the control standard solutions minus those in the equilibrium solutions at the end of the batch equilibration period. Phosphorus sorption was characterized using the Langmuir model:

$$C_e/S = C_e/b + 1/K_b$$

where C_e = equilibrium solution-phase P concentration (mmol L^{-1}), S = sorbed-phase concentration (mmol kg^{-1} soil), b = sorption maximum (mmol kg^{-1} soil), and K_b = constant related to bonding energy (L mmol^{-1}).

RESULTS AND DISCUSSION

Efficacy and Mechanisms of Dissolved Reactive Phosphorus Immobilization by Metal By-Products

The noncomposted manure averaged 2034 mg P kg^{-1} , representing about 11% of manure total P content as DRP forms (Table 2). These concentrations of dissolved P have raised environmental concerns about surface applications of manure without incorporation, as is the case with manure applications to permanent pastures

Table 2. Effects of two mineral by-products on dissolved reactive phosphorus (DRP) and the combined (0.5 M NH_4F -extractable P [FEP] + 0.1 M NaOH -extractable P [SHEP]) fractions of non-composted poultry manure.

Treatment	DRP	Sum of
		FEP + SHEP
	mg P kg^{-1}	
Manure alone	2034a†	4950
Al-WTR + manure‡	651b	5125
Fe-rich by-product + manure	206c	5795
LSD _{0.05}	336	1335

† Treatment means followed by same letter are not significantly different at the 0.05 level of probability.

‡ Al-WTR, aluminum-water treatment residual.

or no-till croplands (Sharpley, 1997; Kuykendall et al., 1999). The addition of the two mineral by-products significantly decreased soluble P concentrations of the manure, to less than 651 and 206 mg DRP kg^{-1} by the Al residual and the Fe-rich by-product, respectively. Although on a mole per mole basis the Al-WTR was relatively less effective in reducing manure DRP than the Fe-rich by-product, it contained Al- and Fe-oxides and other amorphous compounds that sorbed and immobilized P in water-insoluble forms in the treated manure.

The reductions in DRP fraction were accompanied by reductions in the fluoride-extractable phosphorus (FEP) fractions in treated manure, compared with the untreated manure (Fig. 1A). While the FEP decrease induced by the Al-WTR was small, the Fe-rich by-product reduced FEP by a factor of 1.6. Expressed as relative concentrations that were normalized against total P (P/P_0), the untreated manure FEP fraction decreased from 0.25 g g^{-1} to 0.21 and 0.14 g g^{-1} for the Al- and Fe-treated manure, respectively (Fig. 1A). When both FEP and NaOH-extractable phosphorus (SHEP) fractions were considered together, the amounts of FEP + SHEP from untreated and treated manure were about equal (Table 2 and Fig. 1). The Fe-rich by-product apparently caused the dissolution of the FEP fraction of the original manure and formed more SHEP compounds. The Fe-rich by-product increased the SHEP from 0.014 to 0.138 g P g^{-1} in the Fe-treated manure. Meanwhile, the Al-WTR did not significantly change the SHEP of the Al-WTR-treated manure though it contained a large amount of iron (Table 1).

Differential affinity for $\text{PO}_4\text{-P}$ existed between the two metal-rich by-products. Attempts at measuring $\text{PO}_4\text{-P}$ sorption isotherms failed as the Fe-rich by-product very effectively immobilized solution P upon hydration of the by-product, resulting in trace P concentration in the equilibrium solution phase for the whole range of standard P solutions. The Al-WTR also showed a high affinity for $\text{PO}_4\text{-P}$ and the P sorption was well described by the Langmuir sorption model (Fig. 2). We derived a S_{max} value of 5.4 mmol kg^{-1} (95% confidence limits = 5.04 and 5.86 mmol kg^{-1}) and a K_b coefficient of 7.1 L mmol^{-1} for the Al-WTR.

The higher affinity for $\text{PO}_4\text{-P}$ by the Fe-rich by-product provided evidence for why the Fe-rich by-product was more efficient in reducing the manure DRP fraction than that of the Al-WTR (Table 2). Hsu (1976) showed that Al^{3+} and Fe^{3+} reacted with and removed PO_4^{3-} from solution following similar precipitation mechanisms. Minimum residual $\text{PO}_4\text{-P}$ concentrations were reduced more effectively with Fe^{3+} than Al^{3+} with a metal to P molar ratio of 1 or greater.

In this study, the greater efficacy of the Fe-rich by-product, added at a 2 to 1 molar ratio of Fe to P, also enhanced the formation of SHEP, even at the expense of the existing FEP of the raw manure (Fig. 1A).

Both by-products increased the citrate-bicarbonate-dithionite extractable phosphorus (CBDP) fraction of treated manure by an average of 0.02 g P g^{-1} (Fig. 1A).

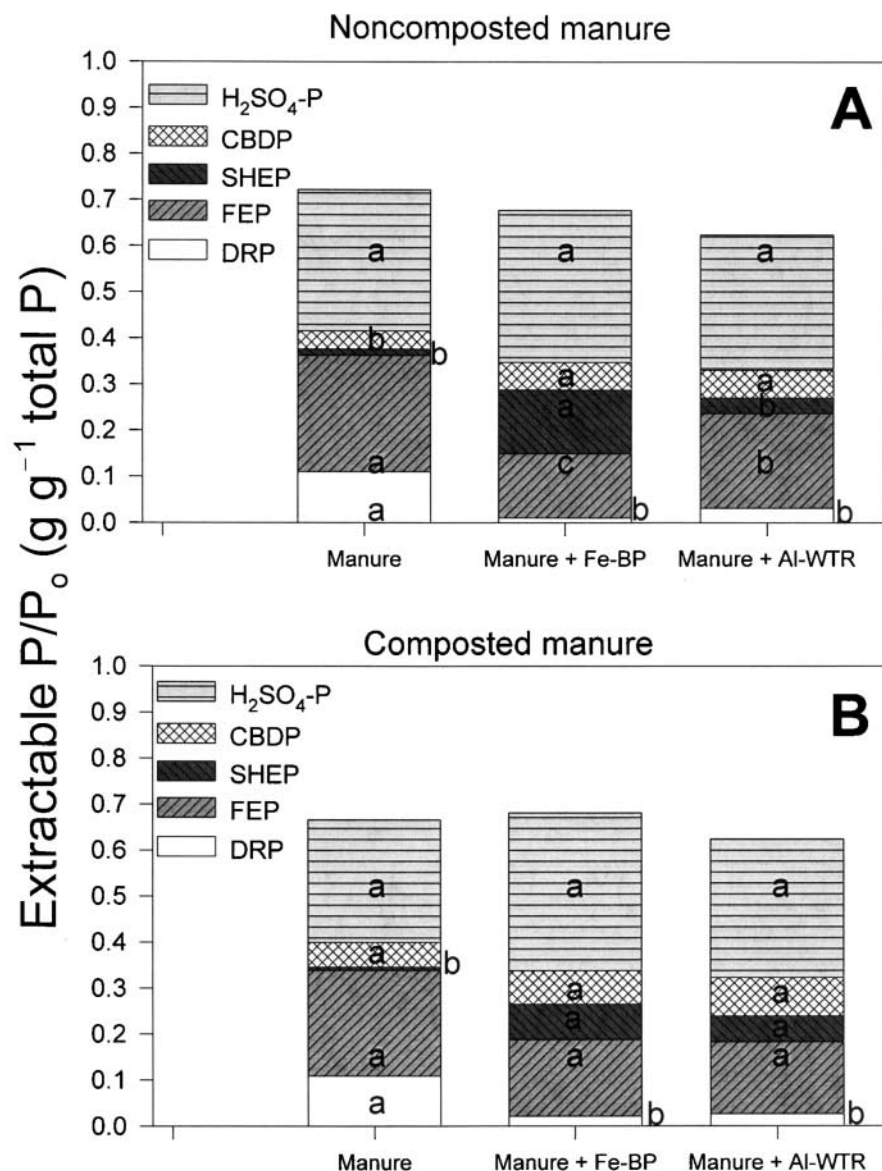


Fig. 1. Effects of Al (Al-WTR) and Fe (Fe-BP) mineral by-products (A) and composting (B) on the distribution of P fractions in poultry manure. Treatment means for each extracted P fraction (DRP, FEP, CBDP, and H₂SO₄-P) followed by the same letter are not significantly different at the 0.05 level of probability. DRP = dissolved reactive P; FEP = 0.5 M NH₄F-extractable P; SHEP = 0.1 M NaOH-extractable P; CBDP = citrate-bicarbonate-dithionite extractable P; and H₂SO₄-P = 0.25 M H₂SO₄-extractable P.

This phosphate pool would be subject to dissolution upon a decline in the reduction-oxidation status of its environment. More reduced conditions would increase the solubility of iron, and in turn the solubility of iron phosphates. The net results would be the dissolution of CBDP and SHEP fractions. The data also showed that there were small but insignificant increases in H₂SO₄-extractable phosphorus (H₂SO₄-P) between untreated and treated manure. And when all extracted fractions were added together, an equal amount of P was extracted from treated and untreated manure, averaging 0.67 g P g⁻¹. Not all of the DRP reduction was accounted for by the increase in the CBDP fraction, most likely the remainder would be immobilized by the most predominant metal in the by-product; that is, the SHEP fraction for the Fe-rich by-product and the FEP + SHEP

fraction for the Al-WTR that contains Al and Fe (Table 1).

Composting Effects

Composting runs lasted from 9 to 20 d. The Al- or Fe-treated manure maintained peak temperature longer than the untreated manure (Fig. 3). The peak temperature of the untreated manure lasted about 8 d and by-product-treated manure lasted for 12 to 15 d. Mass loss reflected the duration of the peak temperatures. The by-product-treated manure lost about 25% of its mass during composting while the untreated manure lost about 10% (Table 3). The mass loss resulted in an enrichment of the P concentration of the composted material and led us to express the changes in size of P pools as relative concentrations, P/P₀.

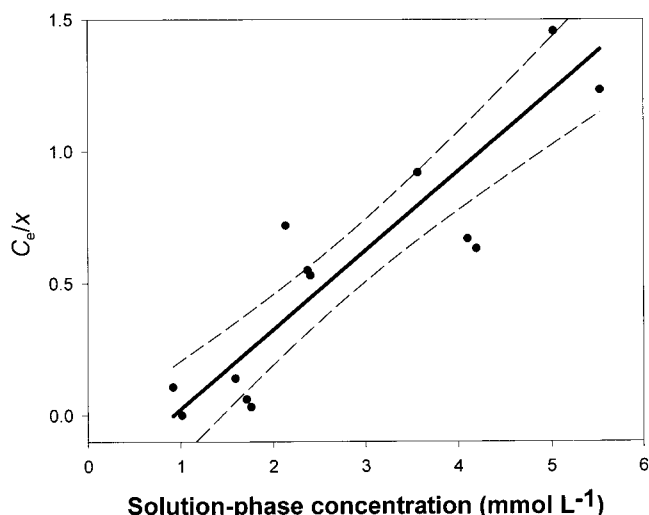


Fig. 2. Sorption of $\text{PO}_4\text{-P}$ on the Al-water treatment residual at 20°C (Langmuir sorption isotherm and 95% confidence intervals).

Composting had no effect on DRP released from untreated poultry manure (Fig. 1). Composting under controlled laboratory conditions did not change the extractability of loosely bound P in the poultry manure. However, this observation was not in agreement with differences in P extractability that were reported for noncomposted and commercially composted beef and dairy cattle manure, and the smaller differences that existed between DRP relative concentrations released by noncomposted and composted poultry manure (Dao, 1999; Sharpley and Moyer, 2000). It was postulated that the high levels and solubility of dietary P supplements (mainly dicalcium phosphate; Table 1) overwhelmingly contributed to the pool of DRP. Composting did not change the solubility of the mineral supplements nor the relative concentrations of extracted DRP.

Composting had no effect on the efficacy of the Al-WTR or Fe-rich by-product in reducing manure DRP relative concentrations. Pooled treatment means showed that Al- and Fe-rich by-products reduced the DRP of noncomposted and composted manure by 39 and 48%, respectively. Significant interactions existed between the effects of mineral by-products and composting for the FEP and SHEP fractions (Fig. 1). Composting did not change the FEP fraction of untreated and Fe-treated manure, but composting decreased significantly the FEP fraction in Al-treated manure (Table 4). There was a corresponding increase in the SHEP fraction. Again, as the Al-WTR contained both Al and Fe compounds, the shift between these two pools may not be surprising.

Composting significantly increased CBDP ($P <$

Table 3. Loss in mass of untreated or by-product-treated poultry manure during composting.

Treatment	Mass loss g g^{-1}
Manure alone	0.095 (± 0.025)†
Al-WTR + manure‡	0.250 (± 0.018)
Fe-rich by-product + manure	0.239 (± 0.013)

† Means and standard deviation ($n = 3$).

‡ Al-WTR, aluminum-water treatment residual.

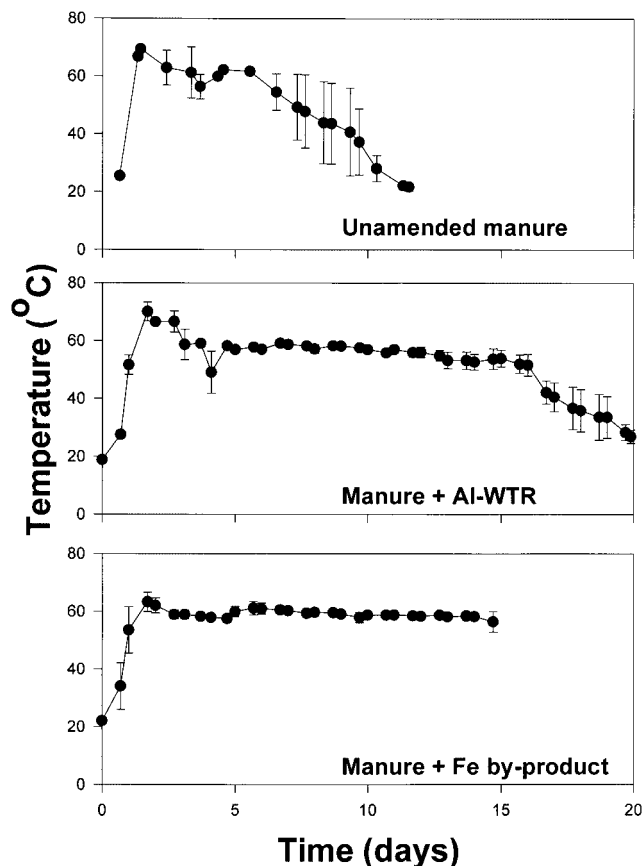


Fig. 3. Compost temperature during the composting of unamended poultry manure and manure amended with Al-water treatment residual (Al-WTR) and iron-rich by-product.

0.0026) for Fe- and Al-treated manure only (Fig. 1). This increase appeared to be at the expense of SHEP for the Fe-treated manure, possibly due to a shift toward more stable crystalline phosphates that subsequently were extracted by the CBDP extractant. In the Al-treated manure, composting increased SHEP and CBDP fractions. Composting promoted the dissolution of FEP forms to form SHEP forms, to a lesser degree similar to the addition of Fe-rich by-product to manure without composting. Composting, in turn, promoted the shift from SHEP to more stable phosphate forms that were extracted next by the CBDP extractant.

Table 4. Effects of composting on the 0.5 M NH_4F -extractable P (FEP) and 0.1 M NaOH -extractable P (SHEP) fractions induced by mineral by-products in poultry manure.

Treatment	Composting	(g g^{-1} total P)	
		FEP	SHEP
Manure	no	0.252 (± 0.001)†	0.013 (± 0.004)
	yes	0.230 (± 0.044)	0.006 (± 0.001)
Fe-rich by-product + manure	no	0.140 (± 0.016)	0.138 (± 0.016)
	yes	0.167 (± 0.012)	0.077 (± 0.019)
Al-WTR + manure‡	no	0.206 (± 0.019)	0.034 (± 0.004)
	yes	0.157 (± 0.021)	0.056 (± 0.004)
LSD _{0.05}		0.029	0.018

† Means and standard deviation ($n = 3$).

‡ Al-WTR, aluminum-water treatment residual.

CONCLUSIONS

The mineral by-products significantly reduced DRP and the FEP fractions of treated manure. Shifts in P pools between FEP and SHEP depended upon the Fe and Al contents of the by-products. The combined FEP + SHEP pool appeared to remain constant. The Fe-rich by-product can cause the dissolution of the FEP fraction in poultry manure. Composting had no significant effect on the immobilization of manure DRP by the mineral by-products but may have enhanced the stabilization of manure P into the CBDP fraction. On the basis of a mole of metal per mole of manure P, the Fe-rich by-product was more efficient in immobilizing dissolved $\text{PO}_4\text{-P}$. Therefore, the choice of immobilization agents affected the eventual stability of immobilized P forms and should be taken into consideration when designing manure treatment methods. The use of relatively inexpensive industrial and municipal by-products containing aluminum and iron to reduce DRP in animal manure prior to land application is a practical manure management practice and promising component of an overall nonpoint-source pollution prevention strategy. In combination with composting to stabilize manure N, mineral by-products can reduce the environmental risks of recycling of manure back into agromonic production.

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